

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Thiophene-2-carbonyl azide

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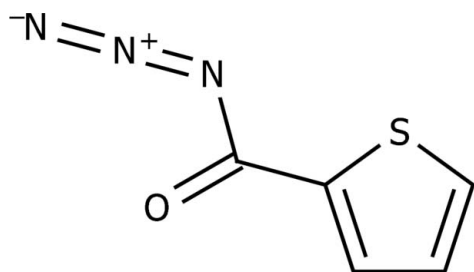
Received 15 July 2013; accepted 16 July 2013

Key indicators: single-crystal X-ray study; $T = 153$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.050; wR factor = 0.154; data-to-parameter ratio = 16.0.

The title compound, $\text{C}_5\text{H}_3\text{N}_3\text{OS}$, is almost planar (r.m.s. deviation for the ten non-H atoms = 0.018 Å) and forms an extended layer structure in the (100) plane, held together *via* hydrogen-bonding interactions between adjacent molecules. Of particular note is the occurrence of $\text{RC}-\text{H}\cdots\text{N}^-=\text{N}^+=\text{NR}$ interactions between an aromatic C—H group and an azide moiety which, in conjunction with a complementary $\text{C}-\text{H}\cdots\text{O}=\text{C}$ interaction, forms a nine-membered ring.

Related literature

For a previous preparation of the title compound, see: Binder *et al.* (1977). For the synthesis of the starting material, 2-thiophenecarbonyl chloride, see: Kruse *et al.* (1989). For related structures, see: Arsenyan *et al.* (2008); Elshaarawy & Janiak (2011); Low *et al.* (2009).



Experimental

Crystal data

$\text{C}_5\text{H}_3\text{N}_3\text{OS}$
 $M_r = 153.16$
 Monoclinic, $C2/c$
 $a = 12.668$ (3) Å
 $b = 6.2153$ (12) Å
 $c = 16.400$ (3) Å
 $\beta = 95.91$ (3)°

$V = 1284.4$ (4) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.43$ mm⁻¹
 $T = 153$ K
 $0.20 \times 0.16 \times 0.15$ mm

Data collection

Nonius KappaCCD diffractometer
 Absorption correction: multi-scan
 (*DENZO* and *SCALEPACK*;
 Otwinowski & Minor, 1997)
 $T_{\min} = 0.920$, $T_{\max} = 0.939$

2728 measured reflections
 1459 independent reflections
 1152 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.154$
 $S = 1.13$
 1459 reflections

91 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.59$ e Å⁻³
 $\Delta\rho_{\min} = -0.49$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C2}-\text{H2}\cdots\text{N1}^{\text{i}}$	0.95	2.63	3.512 (4)	155
$\text{C3}-\text{H3}\cdots\text{N3}^{\text{ii}}$	0.95	2.66	3.396 (4)	135
$\text{C4}-\text{H4}\cdots\text{O1}^{\text{ii}}$	0.95	2.47	3.415 (4)	173

Symmetry codes: (i) $x, y-1, z$; (ii) $x, -y, z+\frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT*; data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2013); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL*, *enCIFer* (Allen *et al.*, 2004) and *publCIF* (Westrip, 2010).

The authors gratefully acknowledge the Robert A. Welch Foundation for their support of GCH *via* the Welch Summer Scholars Program, and Texas Tech University for start-up funds.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5242).

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supplementary materials

Acta Cryst. (2013). E69, o1298 [doi:10.1107/S1600536813019740]

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Comment

The title compound (Fig. 1) displays a different organization in the solid state to that seen in related compounds. It forms one-dimensional hydrogen-bonded chains through the formation of C—H \cdots N/O hydrogen bonds (Table 1 and Fig. 2), that are then linked into two-dimensional sheets in the (1 0 0) plane by further C—H \cdots N interactions. This results in utilization of all the H atoms in the molecule for hydrogen-bonding. All three related structures (Arsenyan *et al.*, 2008; Elshaarawy & Janiak, 2011; Low *et al.*, 2009) are, in contrast, dominated by N—H \cdots O/N hydrogen bonding, resulting in two different one-dimensional chains (Arsenyan *et al.*, 2008; Low *et al.*, 2009), and a two-dimensional sheet (Elshaarawy & Janiak, 2011).

Experimental

The title compound was prepared by the method of Binder *et al.* (1977), from 2-thiophenecarbonyl chloride (Kruse *et al.*, 1989). Crystals suitable for X-ray structure determination were obtained by cooling a toluene solution of the title compound to -30°C.

Refinement

Carbon-bound H atoms were included in calculated positions (C—H distances are 0.95 Å) and refined as riding atoms with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{parent atom})$.

Computing details

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT* (Nonius, 1998); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2013); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008), *enCIFer* (Allen *et al.*, 2004) and *pubCIF* (Westrip, 2010).

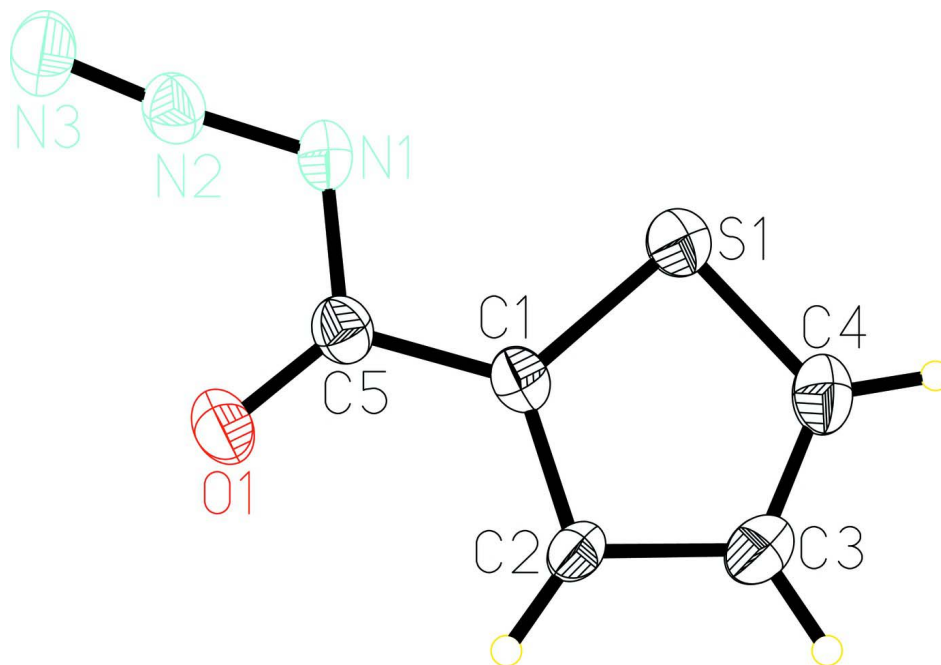


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

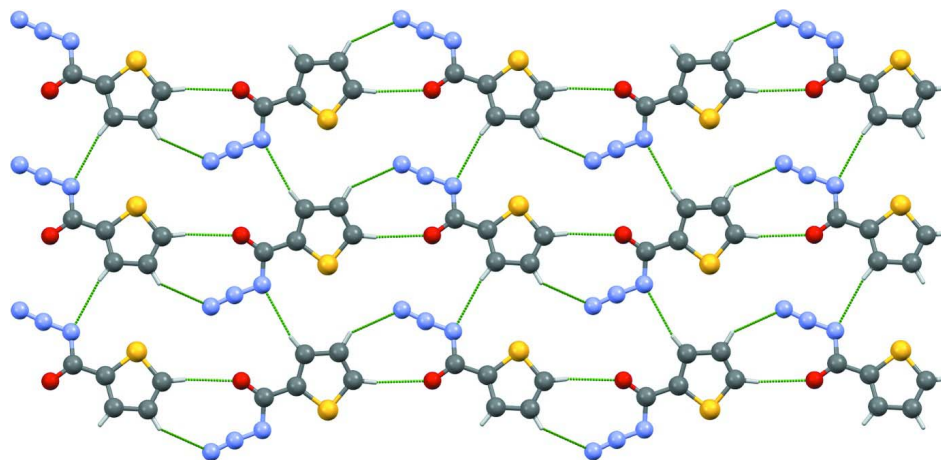


Figure 2

View of one of the hydrogen-bonded sheets in the (1 0 0) plane.

Thiophene-2-carbonyl azide

Crystal data

$C_5H_3N_3OS$

$M_r = 153.16$

Monoclinic, $C2/c$

$a = 12.668 (3) \text{ \AA}$

$b = 6.2153 (12) \text{ \AA}$

$c = 16.400 (3) \text{ \AA}$

$\beta = 95.91 (3)^\circ$

$V = 1284.4 (4) \text{ \AA}^3$

$Z = 8$

$F(000) = 624$

$D_x = 1.584 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1494 reflections

$\theta = 1.0\text{--}27.5^\circ$

$\mu = 0.43 \text{ mm}^{-1}$

$T = 153 \text{ K}$

Block, colourless

$0.20 \times 0.16 \times 0.15 \text{ mm}$

Data collection

Nonius KappaCCD
diffractometer
Radiation source: fine focus sealed tube
 φ and ω scans
Absorption correction: multi-scan
(*DENZO* and *SCALEPACK*; Otwinowski &
Minor, 1997)
 $T_{\min} = 0.920$, $T_{\max} = 0.939$

2728 measured reflections
1459 independent reflections
1152 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\max} = 27.4^\circ$, $\theta_{\min} = 2.5^\circ$
 $h = -15 \rightarrow 16$
 $k = -7 \rightarrow 8$
 $l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.154$
 $S = 1.13$
1459 reflections
91 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0659P)^2 + 3.253P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.59 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.49 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3676 (2)	0.0373 (4)	0.57926 (15)	0.0260 (6)
C2	0.3494 (2)	−0.1827 (4)	0.59864 (15)	0.0238 (6)
H2	0.3401	−0.2988	0.5609	0.029*
C3	0.3478 (2)	−0.1956 (5)	0.68730 (18)	0.0334 (7)
H3	0.3368	−0.3267	0.7150	0.040*
C4	0.3632 (2)	−0.0048 (5)	0.72675 (17)	0.0347 (7)
H4	0.3647	0.0105	0.7845	0.042*
C5	0.3763 (2)	0.1133 (5)	0.49575 (15)	0.0271 (6)
N1	0.3946 (2)	0.3386 (4)	0.49385 (13)	0.0323 (6)
N2	0.39920 (19)	0.4093 (4)	0.42227 (13)	0.0308 (6)
N3	0.4044 (2)	0.4879 (5)	0.36125 (15)	0.0401 (7)
O1	0.36928 (17)	−0.0003 (4)	0.43538 (12)	0.0382 (5)
S1	0.37950 (6)	0.20165 (12)	0.66320 (4)	0.0360 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0249 (13)	0.0295 (14)	0.0237 (12)	−0.0011 (11)	0.0039 (10)	−0.0039 (10)
C2	0.0247 (13)	0.0226 (13)	0.0248 (12)	0.0005 (10)	0.0062 (10)	0.0053 (10)
C3	0.0358 (16)	0.0308 (16)	0.0340 (15)	−0.0005 (12)	0.0055 (12)	0.0079 (12)
C4	0.0364 (16)	0.0438 (18)	0.0240 (13)	0.0034 (14)	0.0035 (11)	0.0026 (12)

C5	0.0251 (13)	0.0311 (15)	0.0255 (13)	−0.0021 (11)	0.0045 (10)	−0.0028 (11)
N1	0.0438 (14)	0.0336 (13)	0.0196 (11)	−0.0007 (11)	0.0028 (9)	−0.0002 (9)
N2	0.0317 (13)	0.0335 (14)	0.0266 (12)	−0.0035 (10)	0.0002 (9)	−0.0026 (10)
N3	0.0461 (16)	0.0450 (16)	0.0286 (13)	−0.0109 (13)	0.0005 (10)	0.0039 (12)
O1	0.0519 (14)	0.0382 (12)	0.0255 (10)	−0.0086 (10)	0.0080 (8)	−0.0072 (9)
S1	0.0487 (5)	0.0318 (4)	0.0275 (4)	−0.0028 (3)	0.0036 (3)	−0.0018 (3)

Geometric parameters (Å, °)

C1—C2	1.428 (4)	C4—S1	1.679 (3)
C1—C5	1.464 (4)	C4—H4	0.9500
C1—S1	1.708 (3)	C5—O1	1.212 (3)
C2—C3	1.459 (4)	C5—N1	1.420 (4)
C2—H2	0.9500	N1—N2	1.260 (3)
C3—C4	1.355 (4)	N2—N3	1.122 (3)
C3—H3	0.9500		
C2—C1—C5	123.1 (2)	C3—C4—S1	113.2 (2)
C2—C1—S1	113.34 (19)	C3—C4—H4	123.4
C5—C1—S1	123.5 (2)	S1—C4—H4	123.4
C1—C2—C3	107.1 (2)	O1—C5—N1	123.7 (2)
C1—C2—H2	126.5	O1—C5—C1	124.8 (3)
C3—C2—H2	126.5	N1—C5—C1	111.5 (2)
C4—C3—C2	114.3 (3)	N2—N1—C5	112.8 (2)
C4—C3—H3	122.8	N3—N2—N1	174.5 (3)
C2—C3—H3	122.8	C4—S1—C1	92.11 (14)
C5—C1—C2—C3	178.9 (2)	S1—C1—C5—N1	−0.6 (3)
S1—C1—C2—C3	−0.5 (3)	O1—C5—N1—N2	2.1 (4)
C1—C2—C3—C4	0.0 (3)	C1—C5—N1—N2	−178.0 (2)
C2—C3—C4—S1	0.5 (4)	C3—C4—S1—C1	−0.7 (3)
C2—C1—C5—O1	−0.1 (4)	C2—C1—S1—C4	0.7 (2)
S1—C1—C5—O1	179.2 (2)	C5—C1—S1—C4	−178.7 (2)
C2—C1—C5—N1	−179.9 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C2—H2 \cdots N1 ⁱ	0.95	2.63	3.512 (4)	155
C3—H3 \cdots N3 ⁱⁱ	0.95	2.66	3.396 (4)	135
C4—H4 \cdots O1 ⁱⁱ	0.95	2.47	3.415 (4)	173

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) *x*, −*y*, *z*+1/2.